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(71) Applicants  
**Kao Soap Co., Ltd.,**  
**1, Nihonbashi-**  
**Kayabacho,**  
**1-chome,**  
**Chuo-ku,**  
**Tokyo,**  
**Japan.**

(72) Inventors  
**Masakatsu Okamoto,**  
**Yoshio Fukui.**

(74) Agents  
**Withers & Rogers,**  
**4, Dyer's Buildings,**  
**Holborn,**  
**London, EC1N 2JT.**

(54) **Soaping agent and soaping method**

(57) Fibrous dyed articles are soaped with a soaping agent comprising a product obtained by adding ethylene oxide to a mixture of a fatty acid triglyceride and a polyhydric alcohol.

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## SPECIFICATION

## Soaping agent and soaping method

5 The present invention relates to a soaping method and agent. More particularly, the present invention relates to a soaping which is conducted in order to remove an unfixed dye when a fibrous article containing polyester fibers has been dyed with a disperse dye. 5

In dyeing fibrous articles containing polyester fibers, a disperse dye is ordinarily used for polyester fibers and an acid dye, a cationic dye, a reactive dye, a basic dye or the like is used for other fibers such as nylon 10 fibers, acrylic fibers, wool fibers and cotton fibers. Dyed articles are ordinarily passed through the soaping step to remove any unfixed dye or paste in the case of printed articles, thereby clarifying the colour pattern, and to improve the feel of various fastnesses. In case of dyed products formed by using a disperse dye, reduction washing is carried out at the soaping step so as to improve various fastnesses. 10

When a disperse dye is used, however, any unfixed dye removed during the soaping operation tends to 15 adhere to the fibers again, resulting in a reduction in the colour clarity of the dyeing. Especially in case of a printed article, if the unfixed dye stains a white portion, the grade of the product is drastically reduced. 15

In order to prevent this staining and improve various fastnesses, there is ordinarily adopted a method in which soaping is carried out in the presence of a surface active agent called "soaping agent".

As the soaping agent, there have heretofore been used ethylene oxide adducts of aliphatic amines, 20 ethylene oxide adducts of nonylphenyl ether, and ethylene oxide adducts of fatty acids. These soaping agents, however, are still insufficient in stain-preventing effect and fastness-improving effect, and therefore, developments of improved soaping agents have been desired in the dyeing art. 20

We have carried out research on this problem and have found that when a soaping agent comprising a product obtained by adding ethylene oxide to a mixture of a fatty acid triglyceride and a polyhydric alcohol is 25 used, re-contamination with an unfixed dye can be sufficiently prevented while improving the fastness of fibrous articles containing polyester fibers satisfactorily. We have now completed the present invention based on this finding. 25

The type of fatty acid triglyceride that is used for the manufacture of the soaping agent of the present invention is not particularly critical. Triglycerides of higher fatty acids are normally used. Examples of 30 suitable fatty acids are behenic acid, stearic acid, oleic acid, linolic acid, palmitic acid, myristic acid, lauric acid and caprylic acid. These higher fatty acids may be used singly or in the form of a mixture of two or more of them. Either natural fatty acid triglycerides or synthetic fatty acid triglycerides may be used. Natural oils and fats, which are natural fatty acid triglycerides, are suitable for carrying out the present invention, because they are easily available. Examples of such natural oils or fats are animal oils and fats such as beef 35 tallow, lard and mutton tallow, and vegetable oils and fats such as coconut oil, palm oil, cotton seed oil, castor oil, rape oil, coconut husk oil, soybean oil, olive oil, linseed oil and corn oil. 35

A compound having 2 to 6 carbon atoms, which contains 2 to 6 hydroxyl groups with the proviso that the number of the hydroxyl groups is equal to or smaller than the carbon number, is preferably used as the polyhydric compound in the present invention. Some Examples are ethylene glycol, propylene glycol, 40 glycerine, 1,2-butylene glycol, 1,3-butylene glycol, 2,3-butylene glycol, 1,2-pentylene glycol, 1,3-pentylene glycol, 2,3-pentylene glycol, 2,4-pentylene glycol, 1,2-hexylene glycol, 1,3-hexylene glycol, 2,3-hexylene glycol, 2,4-hexylene glycol, butane triol, pentane triol, hexane triol, pentaerythritol, sorbitol, mannitol, xylitol and dulcitol. 40

Polyhydric alcohols having 3 carbon atoms are preferred, and glycerol is especially preferred. A mixture of 45 two or more of such polyhydric alcohols may also be used. 45

The HLB value has a significant influence on the properties of the ethylene oxide adduct of the present invention as a soaping agent.

More specifically, it has been found that when the ethylene oxide adduct obtained according to the present invention has an HLB value of from 8 to 14, the properties of the adduct as a soaping agent, that is, the 50 friction fastness and prevention of contamination of a white portion, are greatly improved. Accordingly, in the present invention, the molar ratio of the fatty acid triglyceride to the polyhydric alcohol or the molar ratio of the mixture of the fatty acid triglyceride and polyhydric alcohol is not particularly critical, so far as such molar ratio provides an HLB value of 8 to 14. Ordinarily, however, it is preferred that the polyhydric alcohol be used 0.1 to 5 mols, especially 0.2 to 2 mols, per mol of the fatty acid triglyceride and that ethylene oxide be 55 used in an amount of 1 to 50 mols, especially 5 to 25 mols, per mol of the sum of the fatty acid triglyceride and polyhydric alcohol. 55

The addition reaction method is not particularly critical. Conditions ordinarily adopted for reaction of adding ethylene oxide to active hydrogen-containing compounds may be adopted. More specifically, a catalytic amount of an alkaline substance may be added to a mixture containing a triglyceride and a 60 polyhydric alcohol in the above-mentioned molar ratio, and ethylene oxide is introduced at about 100 to about 200°C under 1 to 5 Kg/cm<sup>2</sup> over a period of several hours to effect reaction. 60

The product obtained by this reaction is a mixture of various compounds, and the composition has not been precisely elucidated. However, it is believed that the main component is a compound in which ethylene oxide is added between the fatty acids constituting the triglyceride and the polyhydric alcohol or glycerine 65 derived from the triglyceride. 65

In the invention, it is practical that the soaping step is conducted like dipping or washing under the following conditions:

- |    |  |    |
|----|--|----|
| 5  | (1) concentration of the soaping agent<br>1 to 5, preferably 2 to 3, g/ℓ     | 5  |
|    | (2) bath ratio of fiber to solution<br>1:10 to 1:60, preferably 1:20 to 1:40 |    |
| 10 | (3) soaping period of time<br>3 to 20, preferably 5 to 15, minutes           | 10 |
|    | (4) bath temperature<br>50 to 90, 60 to 80, °C                               |    |

- |    |   |    |
|----|---|----|
| 15 | The soaping can be carried out effectively either batchwise or continuously.<br>In addition, a reductive washing step may be carried out, using a reducing agent in a desired amount. This amount is preferably 1 to 3 g/ℓ. A preferred reducing agent is a combination of hydrosulfite and caustic soda or thiourea dioxide. | 15 |
| 20 | The present invention will now be described with reference to the following non-limiting Examples:  | 20 |

#### Example 1

- |    |   |    |
|----|---|----|
| 25 | A polyester fabric was printed with a color paste having a composition shown below and preliminarily dried at 100°C for 3 minutes. Then, the fabric was subjected to steaming at 180°C for 3 minutes. The printed cloth was soaped at 80°C for 10 minutes in a bath containing 2 g/ℓ of a soaping agent shown in Table 1, 1 g/ℓ of caustic soda and 2 g/ℓ of hydrosulfite at a bath ratio of 1:30. Then, the printed fabric was washed with running water at 40°C for 5 minutes. The contamination of the white portion and the friction fastness were examined to obtain results shown in Table 1. | 25 |
|----|---|----|

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|----|-----------------------------|----|
| 30 | Composition of Color Paste: | 30 |
|----|-----------------------------|----|

35	Terasil Navy Blue BGL Liquid (disperse dye manufactured by Ciba-Geigy)	8 parts by weight	35
	Terpene	18 parts by weight	
40	Etherified starch (Sorbitose C-5)	5 parts by weight	40
	Citric acid	0.2 part by weight	
	Water	68.8 parts by weight	

- |    |  |    |
|----|--|----|
| 45 | The HLB value was calculated according to the following Griffin's formula: | 45 |
|----|--|----|

$$\text{HLB value} = 20 (1 - S/A)$$

- |    |  |    |
|----|--|----|
| 50 | wherein S stands for the saponification value and<br>A stands for the acid value of the fatty acid used. | 50 |
|----|--|----|

In this case, the saponification value of the reaction mixture was calculated by dividing the saponification value of the triglyceride by the ratio of the weight increase by the polyhydric alcohol and ethylene oxide, and the acid value of the fatty acid formed by decomposition of the triglyceride was adopted as the acid value (see "Handbook of Surface Active Agents", page 308, published by Sangyo Tosho Kabushiki Kaisha in 1960).

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|----|--|----|
| 55 | The friction fastness was determined according to the method of JIA L-0849. More specifically, the dyed fabric was rubbed with a white fabric under a certain load, and the stain of the white fabric was measured by a gray scale for determination of the stain (Dyeing Fastness Department of Japan Standard Association) and the friction fastness was determined based on the measured stain. | 55 |
|----|--|----|

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|----|---|----|
| 60 | The contamination degree of a white portion was determined by treating a white fabric together with the dyed fabric in the same soaping bath and measuring the stain of the white fabric by a gray scale for determination of the stain (Dyeing Fastness Department of Japan Standard Association). | 60 |
|----|---|----|

TABLE 1

Run No.	Triglyceride			Soaping Agent		Mol number of Added Ethylene Oxide	HLB Value	Friction Fastness	Contamination Degree of White Portion
	Kind	Mol	Kind	Polyhydric Alcohol	Mol				
1 (product of present Invention)	beef tallow	1	glycerine	1	10.0	10.9	4-5	4	
2 (ditto)	ditto	1	ditto	1	12.0	11.6	5	4-5	
3 (ditto)	ditto	1	ditto	1	15.0	12.9	5	4	
4 (ditto)	ditto	1	sorbitol	1	12.0	11.5	5	4-5	
5 (ditto)	rape oil	1	glycerine	1	12.0	11.7	5	4-5	
6 (ditto)	ditto	1	ditto	1	3.5	6.7	4-5	3-4	
7 (ditto)	ditto	1	ditto	1	4.9	8.0	4-5	4	
8 (ditto)	ditto	1	ditto	1	8.1	10.0	5	4-5	
9 (ditto)	ditto	1	ditto	1	15.0	12.6	5	4-5	
10 (ditto)	ditto	1	ditto	1	20.0	13.7	5	4	
11 (ditto)	ditto	1	ditto	1	25.0	14.6	5	3-4	
12 (ditto)	ditto	1	ethylene glycol	1	10.0	10.7	5	4-5	
13 (ditto)	ditto	1	ditto	0.5	15.0	11.1	5	4-5	
14 (ditto)	ditto	1	ditto	1.5	16.0	13.8	5	4	
15 (ditto)	ditto	1	pentane triol	1	10.0	11.0	5	4-5	

TABLE 1 (continued)

Run No.	Triglyceride			Soaping Agent		Polyhydric Alcohol	Mol Number of Added Ethylene Oxide	HLB Value	Friction Fastness	Contamination Degree of White Portion
	Kind	Mol	Kind	Mol	Mol					
16 (product of Present Invention)	rape oil	1	pentane triol	0.5	15.0	11.3	5	4-5		
17 (ditto)	soybean oil	1	glycerine	1	10.0	11.2	5	4-5		
18 (ditto)	coconut oil	1	ditto	1	15.0	13.8	5	4		
19 (ditto)	rape oil	1	ditto	2	12.0	13.3	5	4		
20 (ditto)	ditto	1	ditto	0.5	12.0	10.4	5	4-5		
21 (ditto)	ditto	1	ditto	0.2	12.0	9.4	4-5	4		
22 (Conventional product)	adduct of 9 mols of ethylene oxide to nonylphenyl ether					12.8	4	3		
23 (ditto)	adduct of 9 mols of ethylene oxide to oleic acid					11.5	4	3-4		
24 (ditto)	adduct of 15 mols of ethylene oxide to oleyamine					14.3	4	3		
25 (ditto)	sodium $\alpha$ -olefin-sulfonate (having 16-19 carbon atoms)						4-5	3		

*Example 2*

The treatment was carried out in the same manner as described in Example 1 except that Kayalon Polyester Navy Blue 2G-SF Paste (manufactured by Nippon Kayaku Kabushiki Kaisha) was used instead of the dye used in Example 1. The obtained results are shown in Table 2.

TABLE 2

Run No.	Soaping Agent			Mol number of Added Ethylene Oxide	HLB Value	Friction Fastness	Contamination Degree of White Portion
	Triglyceride Kind	Mol	Polyhydric Alcohol Kind				
26 (product of present invention)	rape oil	1	glycerine	1	7.0	8.9	4-5
27 (ditto)	cocohut oil	1	ditto	1	10.0	11.7	4-5
28 (ditto)	beef tallow	1	sorbitol	1	12.0	11.5	5
29 (ditto)	linseed oil	1	glycerine	1	11.0	11.4	5
30 (conventional product)	adduct of 9 mols of ethylene oxide to nonylphenol ether					12.8	3
31 (ditto)	adduct of 9 mols of ethylene oxide to oleic acid					11.5	3-4

**Example 3**

Equal amounts of a polyester fabric dyed according to the method of Example 1 and a cotton fabric dyed under the following conditions were soaped at 80°C for 10 minutes at a bath ratio of 1 : 30 in a soaping bath containing 2 g/l of a soaping agent shown in Table 3 and 2 g/l of soda ash. Then, the fabrics were washed with running water at 40°C for 5 minutes, and the friction fastness and the contamination degree of a white portion were determined with respect to each fabric. The obtained results are shown in Table 3.

**Composition of Color Paste:**

10	Kayacion Turquoise Blue P-CF (reactive dye)	3 parts by weight	10
	Urea	8 parts by weight	
15	Sodium alginate (5%)	50 parts by weight	15
	Sodium m-nitrobenzene-sulfonate	1 part by weight	
20	Sodium bicarbonate	2.5 parts by weight	20
	Water	34.5 parts by weight	

**Printing Conditions:**

After printing, the fabric was preliminarily dried at 80°C for 5 minutes, subjected to steaming at 130°C for 5 minutes and then soaped.

**TABLE 3**  
**Soaping Agent**

Run No.	Soaping Agent				HLB Value
	Triglyceride		Polyhydric Alcohol		
	Kind	Mol	Kind	Mol	Mol Number of Added Ethylene Oxide
32 (present invention)	beef tallow	1	glycerine	1	10.0
33 (ditto)	ditto	1	ditto	1	12.0
34 (ditto)	ditto	1	ditto	1	15.0
35 (ditto)	coconut oil	1	ditto	1	15.0
36 (ditto)	rape oil	1	ditto	1	7.0
37 (ditto)	ditto	1	ditto	1	12.0
38 (conventional product)	adduct of 9 mols of ethylene oxide to nonylphenyl ether				12.8
39 (ditto)	adduct of 9 mols of ethylene oxide to oleic acid				11.5

TABLE 3 (continued)

	Run No.	Polyester		Cotton		
		Friction Fastness (dry)	Contamination Degree of White Portion	Friction Fastness (dry)	Contamination Degree of White Portion	
5						5
10	32 (present invention)	4	4-5	4	4	10
	33 (ditto)	4-5	5	4	4-5	
	34 (ditto)	4-5	4-5	4	4	
15	35 (ditto)	4-5	4-5	4	4	15
	36 (ditto)	4	4-5	4	4	
	37 (ditto)	4-5	5	4	4-5	
20	38 (conventional product)	4	3-4	4	3	20
	39 (ditto)	4	4	4	3	
25						25

*Example 4*

- 30 A polyester/cotton fabric (65/35 weight ratio) was printed with a color paste having a composition shown below, preliminary dried at 80°C for 5 minutes, subjected to hot air fixation at 190°C for 1 minute and then subjected to steaming at 130°C for 3 minutes. The dyed fabric was soaped at 80°C for 10 minutes at a bath ratio of 1 : 30 in a soaping bath containing 2 g/l of a soaping agent shown in Table 4. After soaping, the fabric was washed with running water at 40°C for 5 minutes. The friction fastness and the contamination degree of a white portion were determined. The obtained results are shown in Table 4.
- 35

## Composition of Color Paste:

- Color Paste (1):
- 40 Kayaron Polyester Turquoise 2 parts by weight 40  
Blue GL-S (disperse dye)
- Water 48 parts by weight
- 45 Base paste 50 parts by weight 45  
(5% sodium alginate)
- Color Paste (2):
- 50 Cibaron Turquoise Blue 2 parts by weight 50  
CF-P (reactive dye)
- Urea 8 parts by weight
- 55 Sodium bicarbonate 2.5 parts by weight 55
- Sodium m-nitrobenzene-sulfonate 1 part by weight
- 60 Water 36.5 parts by weight 60
- Base paste 5 parts by weight  
(5% sodium alginate)
- 65 Color pastes (1) and (2) were mixed at a ratio of 1 : 1 before printing. 65



TABLE 4

Run No.	Triglyceride			Soaping Agent			HLB Value	Friction Fastness	Contamination Degree of White Portion
	Kind	Mol		Polyhydric Alcohol	Kind	Mol			
40 (product of present invention)	beef tallow	1		glycerine	1	10.0	10.9	4	4
41 (ditto)	ditto	1		ditto	1	12.0	11.6	4-5	4-5
42 (ditto)	ditto	1		ditto	1	15.0	12.9	4	4
43 (ditto)	coconut oil	1		ditto	1	15.0	13.8	4	4
44 (ditto)	rape oil	1		ditto	1	7.0	8.9	4	4
45 (ditto)	ditto	1		ditto	1	12.0	11.7	4-5	4-5
46 (conventional product)	adduct of 9 mols of ethylene oxide to nonylphenyl ether						12.8	3-4	3
47 (ditto)	adduct of 9 mols of ethylene oxide to oleic acid						11.5	4	3

## CLAIMS:

1. A soaping agent comprising a product obtained by adding ethylene oxide to a mixture of a fatty acid triglyceride and a polyhydric alcohol.
- 5 2. A soaping agent as set forth in Claim 1 wherein the HLB value of the product is 8 to 14. 5
3. A soaping agent as set forth in Claim 1 or 2 wherein the polyhydric alcohol is a compound having 2 to 6 carbon atoms and 2 to 6 hydroxyl groups with the proviso that the number of the hydroxyl groups is not greater than the number of carbon atoms.
4. A soaping agent as set forth in Claim 3 wherein the polyhydric alcohol is glycerol.
- 10 5. A soaping agent as set forth in any of Claims 1 to 4 wherein the fatty acid triglyceride is a natural oil or 10 fat.
6. A method for soaping fibrous dyed articles which comprises dipping and washing the dyed articles in a solution of a soaping agent according to any preceding claim.

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